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STUDIES ON AROMA OF CURED HAM

SUMMARY—Cured and uncured hams, raw, cooked or cooked-smoked, were analyzed for free amino acids. A number of amino acids decreased on curing. Total free amino acid concentration increased on cooking—slightly in uncured hams and to a much larger extent in cured hams. Smoking resulted in negligible change in total amino acid concentration of hams, although a number of individual amino acids were affected. Aqueous extracts and diffusates of cured and uncured hams were differentiated by a trained panel on the basis of aromas produced on heating. Smoke aroma could be detected in cured smoked samples but only with difficulty, if at all, in uncured smoked samples. Precursors of basic meaty aroma are water extractable from all hams examined, whereas components or precursors of cured and smoky aroma may be extracted from hams with chloroform-methanol. Gas chromatography of volatiles developed on heating of ham diffusates and lipid extracts was carried out and the odors of the components separated were observed. Some variations among the patterns of volatiles from the 6 types of hams studied were observed, but no single component had a meaty or cured aroma.

INTRODUCTION

THE CURING of ham has been investigated extensively in the past, with most of the work reported dealing with the formation and changes of color. Many of these reactions have been elucidated (Wilson, 1960; Lawrie, 1966). Cured ham may be easily distinguished from uncured ham on the basis of flavor. However, relatively little is known about the interaction of the cure components with meat components reflected in the modification of meat flavor due to curing.

Only a few reports dealing with flavor constituents of cured hams are available (Lillard et al., 1969; Ockerman et al., 1964; Cross et al., 1965). These indicate that cured ham flavor is found in the volatiles obtained by vacuum distillation of ham. Many of the volatile components of cured ham thus far identified have also been found in uncured ham as well as in other types of meat (Landmann et al., 1966; Solms, 1968). There has been no indication that any specific component or components of cured ham flavor possess the characteristic "cured" aroma.

According to Lillard et al. (1969) the flavor compounds and flavor precursors of country-cured hams were not water soluble. On the other hand, Baker (1961) reported that the freeze-dried water-soluble fraction of fresh pork yielded a strong ham flavor after curing and cooking. A variety of techniques for the curing of hams is being used in the food industry today. Consequently, "cured flavor" has not been clearly defined, and some variations in cured flavor may be attributed to the method of preparation.

In the present work we have studied various fractions of ham to isolate and identify the cured-ham aroma and to follow the changes occurring in pork during curing, cooking and smoking. In particular, analysis of the free amino acids in hams was undertaken as a possible indication of the processes leading toward the formation of ham flavor.

EXPERIMENTAL

Preparation of hams

The study involved 18 hams. The samples were prepared in 3 sets of 6 hams consisting of uncured raw (UR); uncured cooked (UC); uncured cooked and smoked (US); cured raw (CR); cured cooked (CC) and cured cooked and smoked (CS).

Raw hams and raw, cure-pumped hams, average weight 6 kg each, were obtained from a commercial source and aged 48 hr before processing. All samples referred to as cooked were heated in a Model ESQ-1 SH Smokehouse (Drying Systems Co., Div. of Michigan Oven Co.) without smoke at 120°F for 2 hr, 140°F for 2 hr, 160°F for 4 hr, 180°F for 2 hr and 190°F for 1.5 hr. The internal temperature of all hams at the end of this heating program was 150°F. The same heating program was used in preparing the smoked samples with the addition of smoke generated from commercial sawdust using a Meat Packers of America Junior Model Smoke Generator. The samples were smoked for 9.5 hr with a 2-hr drying-off period without smoke at the beginning of the heating program.

Preparation of aqueous extracts of ham

Steaks ½-in. thick were taken from each ham for organoleptic evaluation. After removal of as much visible fat as possible the remainder of the lean meat in each ham was ground twice to ensure a homogeneous mixture for analyses. 200 g of ground ham were homogenized with 200 ml of cold deionized water for 2 min in a blender. The mixture was centrifuged at 6,870 × g and the insoluble residue extracted twice with 100 ml of water each time. The combined supernatants, cooled to 2°C, were filtered through glass wool to remove suspended fat.

Preparation of ham diffusates

An aliquot of the aqueous extract (350 ml) was dialyzed against 350 ml of deionized water for 64.5 hr with 4 changes of water, using 7.5-cm cellulose casing (Union Carbide Co.) previously washed with water to remove glycerin. The dialysis procedure was carried out at 4°C. The combined diffusates (1,750 ml) were lyophilized, dissolved in deionized water to a final volume of 100 ml and stored at −18°C.

Preparation of chloroform-methanol extracts of ham

Lipid fractions of the ground hams were prepared by the extraction procedure of Folch et al. (1957).

100 g of meat were homogenized with 500 ml of chloroform-methanol (2:1, v/v) for 3 min in a blender. The slurry was filtered through Whatman No. 1 filter paper previously washed with the solvent and the residue extracted with an additional 500 ml of chloroform-methanol. The combined extracts were evaporated in vacuo at 30–35°C after washing with 200 ml of water to remove traces of water-soluble material. Extracts of uncured samples were pale amber in color and yielded an amber oil on removal of solvent. Extracts of cured samples were wine red in color and on evaporation yielded an amber oil containing particles of dark-brown pigment in suspension.

Analytical methods

Analyses for amino acids and other ninhydrin-reactive compounds were performed according to Spackman et al. (1958) using a Phoenix automatic amino acid analyzer.

Qualitative analyses of ham diffusates were carried out by thin-layer chromatography on Eastman Chromagram sheets 6064 (cellulose) and 6061 (silica gel) using the solvent systems: (A) n-propanol-ammonia (70:30 v/v) and (B) formic acid-butanone-t-butanol-water (15:30:40:15 v/v). Compounds on chromatograms were detected as described previously (Zaika et al., 1968).

Gas chromatography

All samples were heated in a Loenco Model 260 pyrolyzer and the volatile compounds introduced directly into the chromatographic column of an F and M Model 810 gas chromatograph by means of a valve. A 6-ft by ¼-in. stainless steel column packed with 15% Carbowax 20M TPA on 60-80-mesh Gaschrom P was used with helium flow of 63 ml/min. Column temperature was maintained at 70°C for 5 min, then programmed at 8°/min to 180°C and held at that temperature for a total analysis time of 60 min. The column effluent stream was split before passage into the flame ionization detector to permit evaluation of odors of components eluted from the column. Ham diffusate (0.5 ml) was used for analysis. The sample solution was introduced into a pyrex U-tube 8 cm long and 0.4 cm id and lyophilized. The dry material was heated in the pyrolyzer unit at an oven temperature of 250°C for 2 min and injected into the column for 2 min. Lipid fractions were treated in a similar manner.

Odor evaluation

Aroma was developed by heating 1.0 ml of

Table 1—Panel evaluations of odors obtained on heating ham extracts and diffusates.

	Extracts						Diffusates					
	UR	UC	US	CR	CC	CS	UR	UC	US	CR	CC	CS
Smoky			(√)			√						√
Chalky							√	√	√	√	√	√
Brothy	√	√	√	√	√	√	√	√	√	√	√	√
Cured				√	√	√				√	√	√
Salty				√	√	√						
Sweet				√	√	√				√	√	√
Porky	√	√	√									
Ham				√	√	√				√	√	√
Roast meat	√	√	√	√	√	√	√	√	√			
Burnt							√	√	√	√	√	√

aqueous sample or 0.1 ml of lipid fraction in a 10-ml beaker at 250°C on a calibrated hotplate. The aromas formed at different stages were noted, i.e., on warming, during boiling, at or just before dryness, at browning and after browning. A 5-member panel was trained over a period of 3 wk to recognize the aromas produced on heating and pyrolyzing whole pieces of ham and the diffusates of water extracts of ham. A standardized vocabulary was developed for the aromas of each type of preparation. After describing the aromas of unknown samples, differences in panelists' descriptions were resolved by referring to the standard preparations. Difficulties arose in obtaining uniform descriptions, because the odors change continuously during heating and the times of sniffing by the panelists were not synchronized. Furthermore, differences in heating rates occurred as a result of variations in temperature among the hotplates and variations in thickness of the beaker glass.

RESULTS & DISCUSSION

CURED and uncured hams could be identified readily by taste and odor on pan frying. All of the uncured ham steaks (UR, UC, US) were judged to have the odor and taste of pork chops or roast pork. The flavor of cured samples (CR, CC, CS) was identified with ham. Smoke was readily detected in CS samples. Only faint smoke flavor was detected in the outside portion of the US ham samples and none on the inside portion of the ham steaks.

In the present work it was found that aqueous extracts of cured and uncured hams (with or without smoking) could be identified by heating and observing the aromas produced (Table 1). For smoke-treated samples the smoke odor was quite noticeable in the cured ham extracts but could not be identified with certainty in uncured samples. The smoke aroma was evident at the beginning of the heating period and probably consisted of easily volatilized compounds dissolved in the extract.

When the aqueous extracts of hams were dialyzed, the diffusates from the cured hams (CR, CC, CS) gave, on heating, a cured-meat aroma, whereas the diffusates from uncured hams (UR, UC, US) gave an aroma of cooked meat (Table 1). The dialysate fractions containing the higher molecular weight components yielded only faint noncharacteristic odors on heating. Differences in odor of cured and uncured extracts (or diffusates) were most noticeable during the boiling stage. On heating to dryness (browning) the odors obtained were essentially those of roast meat for all of the samples, with the cured samples having sweeter aromas (possibly due to compounds originating from added sugar in the cure solution).

Qualitative analysis of ham diffusates was carried out by thin-layer chromatography. Not many differences were noted between cured and uncured samples. Cured samples contained a large amount of an iodine-reactive compound as shown on the thin-layer chromatogram. This material also reacted with bromphenol blue-methyl red indicator, which is characteristic of a basic compound. Creatinine content increased to some extent in the cooked samples as compared to the raw. This was in agreement with results obtained by Macy (1966). No significant differences in the purine derivatives (inosinic acid, inosine, hypoxanthine) were noted.

Amino acid analysis of ham diffusates is shown in Table 2. Ratios for each amino acid were calculated (Table 3) to determine whether there are any effects on these compounds due to curing, cooking or smoking. A number of differences in the ratios of free amino acids of the 6 types of hams were noted. The difference was considered meaningful if it was greater that 20% (an arbitrary value) of the denominator values in Table 3.

Curing resulted in a 26% decrease of total free amino acids of raw hams. A decrease of free amino acids in meat due to curing is not unexpected. These could be converted to α-hydroxy acids by way of the VanSlyke reaction, in which the amino group may interact with nitrous acid formed from sodium nitrite in the cure. There was a significant reduction of

Table 2—Free amino acids[1] and nitrogenous compounds in hams.

	UR	CR	UC	CC	US	CS
Taurine	2.92	2.40	3.66	4.32	3.73	3.78
Urea	2.39	1.48	2.05	2.16	4.09	5.13
Aspartic Acid	0.068	0.29	0.11	0.087	0.12	0.084
Threonine	0.50	0.34	0.46	0.47	0.52	0.56
Serine	0.68	0.56	0.84	0.69	0.82	0.90
Glutamine[2]	0.99	0.54	1.17	1.36	0.59	0.58
Proline	0.45	0.36	0.55	0.46	0.58	0.61
Glutamic Acid	0.75	0.45	1.21	0.94	1.38	1.12
Glycine	1.76	1.04	2.20	1.51	1.95	1.87
Alanine	3.22	1.96	3.72	3.01	3.35	3.66
Valine	0.35	0.45	0.65	0.54	0.66	0.48
Methionine	0.16	0.10	0.18	0.18	0.18	0.16
Isoleucine	0.24	0.24	0.36	0.38	0.40	0.34
Leucine	0.47	0.44	0.73	0.65	0.74	0.64
Tyrosine	0.23	0.22	0.30	0.29	0.32	0.27
Phenylalanine	0.22	0.18	0.29	0.28	0.33	0.26
Ornithine	0.068	0.031	0.050	0.035	0.11	0.43
Ethanolamine	0.12	0.046	0.14	0.066	0.10	0.81
Ammonia	2.43	1.91	3.09	4.46	3.38	3.11
Lysine	0.38	0.38	0.59	0.58	0.58	0.59
Histidine	0.16	0.12	0.26	0.26	0.32	0.34
Anserine	0.33	0.21	0.45	0.40	0.52	0.27
Carnosine	10.86	8.21	12.31	12.01	12.25	10.11
Total	29.75	21.96	35.37	35.14	37.02	36.10

[1] μmoles/g meat.

[2] Includes asparagine.

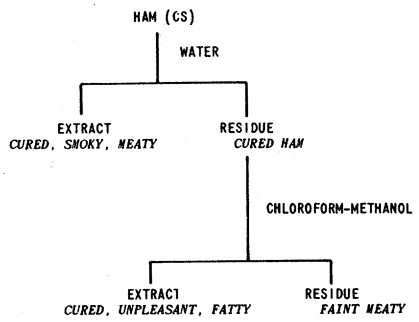

Fig. 1–Extraction of ham flavor.

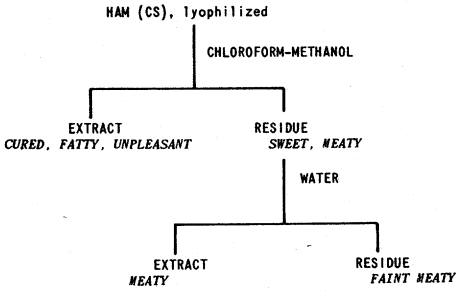

Fig. 2–Extraction of ham flavor.

Table 3–Ratios of free amino acids and nitrogenous compounds in hams.

	UR/CR	UR/UC	CR/CC	UC/US	CC/CS	UC/CC	US/CC
Taurine	1.22	0.80	0.56	0.98	1.14	0.85	0.99
Urea	1.62	1.17	0.68	0.50	0.42	0.95	0.80
Aspartic Acid	0.24	0.64	3.29	0.90	1.04	1.22	1.40
Threonine	1.46	1.06	0.73	0.89	0.84	1.00	0.93
Serine	1.21	0.81	0.81	1.01	0.76	1.21	0.91
Glutamine	1.82	0.84	0.40	1.20	2.36	0.86	1.02
Proline	1.23	0.82	0.80	0.95	0.75	1.20	0.94
Glutamic Acid	1.65	0.62	0.48	0.87	0.83	1.29	1.23
Glycine	1.69	0.80	0.69	1.12	0.81	1.46	1.04
Alanine	1.64	0.87	0.65	1.11	0.82	1.23	0.92
Valine	0.78	0.54	0.84	0.99	1.13	1.21	1.38
Methionine	1.51	0.89	0.57	1.01	1.12	0.97	1.08
Isoleucine	0.99	0.67	0.65	0.91	1.09	0.96	1.15
Leucine	1.05	0.64	0.69	0.98	1.02	1.12	1.16
Tyrosine	1.05	0.79	0.76	0.93	1.08	1.01	1.16
Phenylalanine	1.24	0.75	0.63	0.89	1.06	1.04	1.24
Ornithine	2.19	1.36	0.88	0.46	0.08	1.42	0.25
Ethanolamine	2.59	0.85	0.70	1.33	0.08	2.13	0.13
Ammonia	1.27	0.79	0.43	0.91	1.43	0.69	1.09
Lysine	0.97	0.63	0.66	1.02	0.99	1.02	0.99
Histidine	1.31	0.62	0.48	0.83	0.76	1.01	0.92
Anserine	1.57	0.73	0.53	0.87	1.47	1.14	1.93
Carnosine	1.32	0.88	0.67	1.00	1.19	1.02	1.21
Total	1.35	0.84	0.63	0.96	0.97	1.01	1.03

most of the amino acids in CR samples as compared to UR samples, particularly urea, threonine, glutamine, glutamic acid, glycine, alanine, methionine, ornithine, ethanolamine and anserine. However, aspartic acid and valine increased.

Cooking generally resulted in an increase of free amino acids in hams. The total free amino acid content increased 19% for uncured and 60% for cured hams compared to the corresponding raw hams. Carnosine, the major amino constituent derived from cured as well as uncured ham, accounted for 26 and 29% of total increases on cooking in uncured and cured hams, respectively. Taurine, glutamic acid, glycine, iso-leucine, leucine, tyrosine, phenylalanine, ammonia, lysine, histidine and anserine increased both in cured and uncured hams. Valine and aspartic acid also increased in uncured hams but ornithine decreased. More extensive changes due to cooking were noted for amino acids in cured hams. Thus, the contents of urea, threonine, glutamine, proline, alanine, methionine and ethanolamine were higher in CC samples than in CR samples. Aspartic acid was, however, decreased in the cured samples on cooking. The total levels of amino acids in cured and uncured cooked hams were comparable (UC total/CC total = 1.01), although there were variations in individual amino acids. Amino acids, important constituents of the water-soluble fraction of meat, probably contribute toward formation of meat flavor on heating by interaction with carbohydrates and degradation into volatile compounds and browning products. Macy et al. (1964) reported that amino acids in pork diffusate decreased 20% on heating at 100°C for 1 hr. Larger decreases were found for beef and lamb diffusates. However, according to Usborne et al. (1968), cooking of pork l. dorsi muscles to an internal temperature of 77°C resulted in a general increase of free amino acids, presumably due to degradation of proteins. In a similar study with beef, Macy (1966) reported that amino acids increased on cooking except threonine, serine, glutamic acid, histidine and arginine, which decreased. On the other hand, Zoltowska (1967) reported that in pork l. dorsi samples, canned and cooked at 95, 103 and 121°C, the free amino acid content decreased as compared to raw samples, particularly under high temperature.

In our study the over-all increase in amino acids observed on cooking may be due to protein hydrolysis caused by heating. Since the hams were subjected to rather low internal cooking temperature (66°C), destruction of amino acids originally present in raw ham through reactions with carbonyl compounds may not have been significant.

Table 4–Some volatile components from ham fractions.

R_t (min)	Uncured ham	Cured ham
Diffusates		
12.0		Small, buttery, sweet
15.3	Small, acidic	Large, acidic
19.0	Small, green	Large, green, haylike
21.0	Large, greasy, nutty	Small, greasy
Lipid extracts		
12.9	Medium, earthy, plantlike	Medium, waxy, aromatic
13.4	Large[1], moldy	
14.5	Large[2], greasy, rotten	Large[2], greasy, rotten
18.6	Small, haylike, cheesy	Large, oily, sweet
20.5	Small, stale fat	Large, pleasant, pastry

[1]Raw samples.
[2]Cooked and cooked-smoked samples.

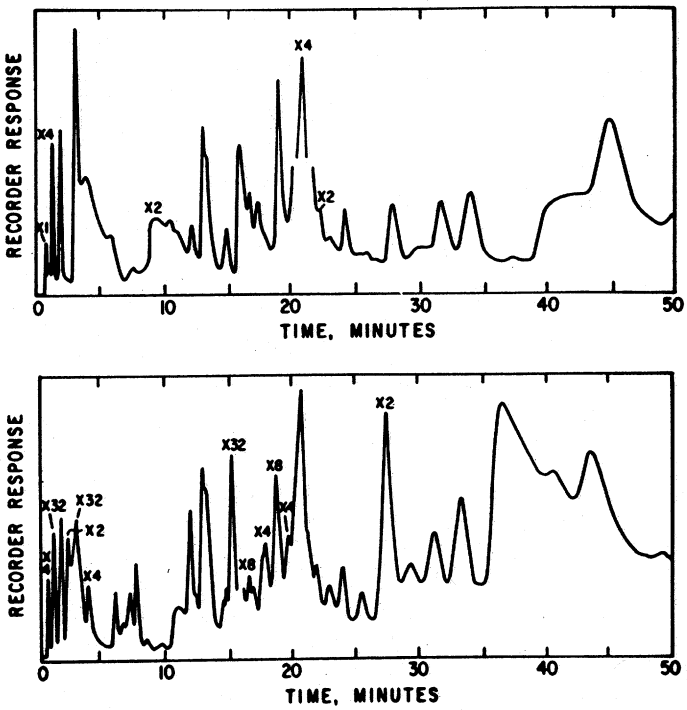

Fig. 3—Gas chromatogram of volatiles from (top) uncured cooked ham diffusate, and (bottom) cured cooked ham diffusate.

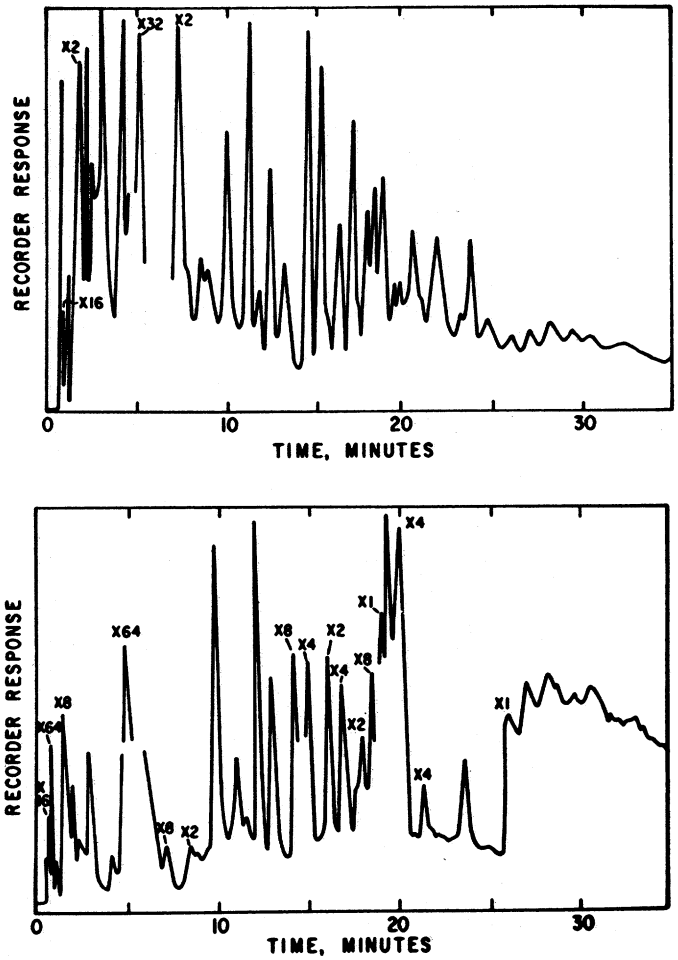

Fig. 4—Gas chromatogram of volatiles from (top) uncured cooked ham lipid extract, and (bottom) cured cooked ham lipid extract.

The effect of smoking on the free amino acids of ham was not so extensive as that of curing or cooking. The content of total free amino acids in smoked hams was 5% higher in uncured and 3% higher in cured hams compared to the corresponding cooked hams. Total levels of amino acids in US and CS hams were quite similar. However, meaningful differences were found for a number of individual amino acids. In uncured samples there was an increase on smoking in urea and orthinine and a decrease in glutamine and ethanolamine. In the cured samples urea, serine, proline, ornithine, ethanolamine and histidine were increased; glutamine, ammonia and anserine were decreased.

Free amino acids in hams have not been studied extensively. Grau et al. (1958) found, qualitatively, higher concentrations of free amino acids in cured, smoked and smoked-cooked meat as compared to fresh meat. Lillard et al. (1969), using a gas chromatographic method, found a much higher level of a number of amino acids in country-cured hams from various commercial sources than in fresh hams. Most of the studies reported probably include aging effects which would lead to protein degradation and a resulting increase in free amino acids in cured hams. Consequently, at present it is difficult to draw any comparison between present and past results concerning the effect of curing on the free amino acids in hams.

The water-insoluble residues from the extraction of cured samples still yielded cured ham aromas on heating. The possibility exists, therefore, that cured odor components or precursors are not water soluble and may be associated with the lipid phase.

Ham samples were extracted with a mixture of chloroform-methanol to yield the total lipid fractions. Cured samples could be readily distinguished from uncured samples when the lipid fractions were heated and their odors observed. Smoke odor could also be detected in the lipid fraction of cured smoked ham (CS). Although the aqueous extracts of cured hams yielded cured, hammy odors, these odors were more intense in the chloroform-methanol-soluble fractions, indicating that cured odor components or precursors were essentially in the lipid phase.

To investigate this further, a sample of cured smoked ham (CS) was divided into two parts: One was extracted first with water and then with chloroform-methanol (Fig. 1); the other was extracted first with chloroform-methanol and then with water (Fig. 2). The various fractions thus obtained were submitted to the panel for odor evaluation. Figure 1 indicates that some of the cured flavor can be extracted with water, but the main portion of cured flavor is soluble in chloroform-methanol. When the ham was first extracted with chloroform-methanol, a cured-ham, fatty aroma was obtained on heating. A meaty aroma was obtained from the water extract of the insoluble residue (Fig. 2). The characteristic cure, as well as smoke, odor was not detected. These results indicate that precursors of the basic meaty aroma are water soluble, whereas precursors of the cured-ham flavor are associated with the lipid phase.

The individual components of ham aroma were examined to determine whether significant differences in the patterns of volatiles existed between cured and uncured ham samples and, if possible, to isolate a component having a cured odor. Therefore, volatile compounds derived from ham diffusates and

ham lipid extracts were separated by gas chromatography. As the components emerged from the chromatographic column their odors were observed.

Representative gas chromatograms of volatiles from ham diffusates and lipid extracts are shown in Figures 3 and 4, respectively. In general, the differences between the 6 types of samples were quantitative rather than qualitative. Complex chromatograms were obtained, with 35–40 peaks for the diffusates and 40–50 peaks for the lipid fractions. It was noted that the material in a diffusate was a good representation of meat aroma, since chromatograms of lyophilized diffusate and lyophilized whole ham were quite similar. Very volatile compounds from ham diffusates possessed unpleasant sulfury or amine-like odors and occurred in both cured and uncured samples. Compounds of intermediate volatility, eluting between 10 and 25 min, had the most interesting odors, in many instances quite intense even though they were present in rather small amounts. A number of components were described as having a green, plant-like odor. The patterns of less-volatile compounds (eluted after 25 min) were similar for cured and uncured samples and the odors in this region were described as burnt or charred.

Many of the volatile components derived from the lipid fractions were described as waxy, fatty, fried, green and aromatic. As in the case of the diffusates, no single component had a cured, meaty or smoky odor. In general, more agreeable aromas were found in the volatiles of cured ham lipid fractions than in uncured samples, although the patterns of peaks on the chromatograms did not differ to a large extent. Cooked samples exhibited a higher level of volatiles than did raw samples.

Some of the outstanding features in the pattern of volatiles derived from ham diffusates and ham lipids are summarized in Table 4. We feel that compounds of intermediate volatility are important contributors toward meat flavor as well as better indicators of the differences between cured and uncured hams. Work is now in progress in our laboratory to isolate and identify the constituents of ham aroma and to further elucidate the effects of curing, cooking and smoking on the flavor of ham.

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